

# The Dissociation Pressures of Hydrated Cupric Sulfate at 35 Degrees Centigrade

By  
THOMAS S. LOGAN

A DISSERTATION  
SUBMITTED TO THE BOARD OF UNIVERSITY STUDIES OF THE  
JOHNS HOPKINS UNIVERSITY IN CONFORMITY  
WITH THE REQUIREMENTS FOR THE  
DEGREE OF DOCTOR OF PHILOSOPHY

Baltimore, 1931





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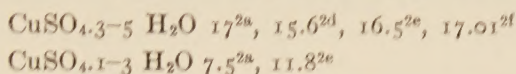
### ACKNOWLEDGMENT

The writer wishes to express here his appreciation to Professors J. C. W. Frazer and W. A. Patrick and to Doctor R. K. Taylor, at whose suggestion and under whose supervision the work described was carried out.



## THE DISSOCIATION PRESSURES OF HYDRATED CUPRIC SULFATE AT 35 DEGREES CENTIGRADE

The dissociation pressures developed by the hydrates of cupric sulfate have been the subject of a number of investigations.<sup>1</sup> Such work has, however, usually been carried out as merely one of a series of determinations involving several salts. The data available at 25° have been tabulated by Wilson,<sup>2</sup> who has pointed out that they are not at all concordant. In the case of hydrated cupric sulfate, the following values at 35° have been published:



The values are in millimeters of mercury. No records of measurements on  $\text{CuSO}_4 \cdot 0-1 \text{ H}_2\text{O}$  at this temperature have been found.

The extreme slowness with which the hydrates of cupric sulfate establish equilibrium pressure has occasioned some comment. Carpenter and Jette<sup>1e</sup> include in their data curves showing that in the case of a mixture of the pentahydrate and tri-hydrates, equilibrium was established from either side only after fourteen days. The influence of small amounts of air on the establishment of true equilibrium has been shown by Patrick and MacGavack.<sup>3</sup> Menzies<sup>1g</sup> discusses this point and describes an experiment whereby he shows that such accumulated air is due to an adsorbed layer on the cupric sulfate which is persistently held.

The purpose of the present work was to prepare a sample of cupric sulfate pentahydrate under air-free conditions, to withdraw the water in successive portions, taking pressure measurements after each withdrawal, and thus to find out how air-free conditions influenced the time required to establish equilibrium.

### Apparatus

An outline of the glass apparatus used is shown in Fig. 1. The hydrated salt was contained in the bulb, A, connected directly to one arm of the manometer. The traps marked a were found necessary to prevent the flowing out of the very fine powder obtained when cupric sulfate is dehydrated. Any powder depositing here could be washed down by condensing water above. The plug, b, made it possible to trap off the hydrate if it was necessary to admit the

<sup>1</sup> Lescoeur: *Ann. Chim. Phys.* (6), 21, 511 (1890); Andreas: *Z. physik. Chem.*, 7, 241 (1891); Foote and Scholes: *J. Am. Chem. Soc.*, 33, 1309 (1911); Frowein: *Z. physik. Chem.*, 1, 5 (1887); Carpenter and Jette: *J. Am. Chem. Soc.*, 45, 578 (1923); Partington: *J. Chem. Soc.*, 123, 160 (1923); Menzies: *J. Am. Chem. Soc.*, 42, 1951 (1920); Schumb: *J. Am. Chem. Soc.*, 45, 342 (1923).

<sup>2</sup> Wilson: *J. Am. Chem. Soc.*, 43, 704 (1921).

<sup>3</sup> Patrick and MacGavack: *J. Am. Chem. Soc.*, 42, 946 (1920).

atmosphere to the system. The arms of the manometer, B, were constructed of tubing with an inside diameter of 12 millimeters. At the base these were run into an air trap which was connected to a length of 1.5 millimeter capillary tubing, sealed through the stopcock, c, to the mercury reservoir, d. This construction was all glass, in order to eliminate any fouling of the mercury by rubber. The height of mercury in the manometer could be regulated by connecting the outlet of the mercury reservoir to a pump.

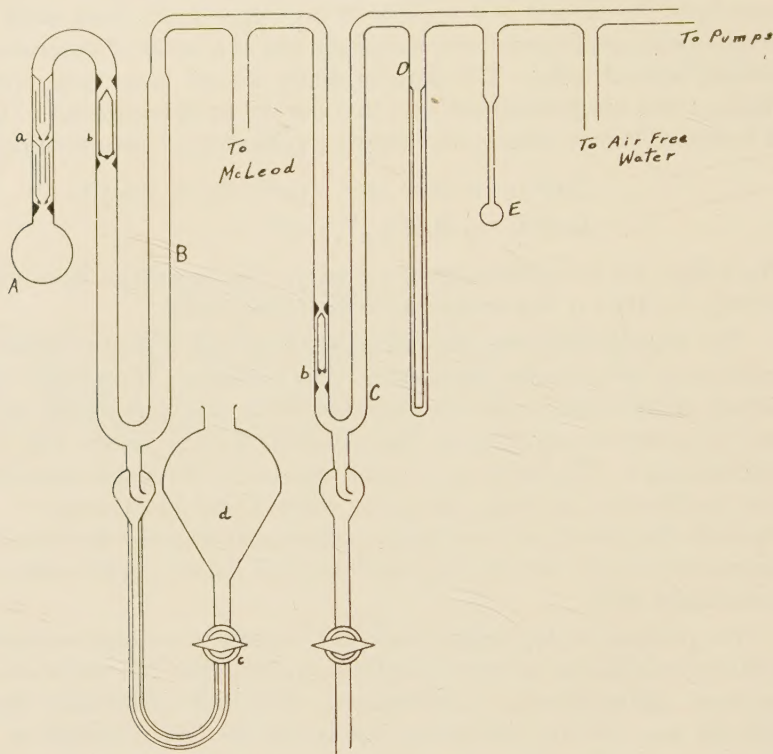


FIG. 1

A McLeod gage was sealed into the manifold as indicated. This was of the type suggested by Taylor.<sup>4</sup> The readings of permanent gas as taken here, when the manometer was drawn down to permit diffusion, could be converted to the actual pressure in the hydrate bulb through a knowledge of the volume ratio involved. In this apparatus this was approximately 5 to 1.

The trap, C, containing another of the plugs, b, served to trap off the system while measurements were being made, in case it was necessary to admit the atmosphere.

A piece of capillary tubing of known cross-section was sealed in at D. By surrounding this with ice it was possible to condense here the approximate amounts of water it was desired to introduce into or remove from the salt.

<sup>4</sup> Taylor: J. Am. Chem. Soc., 50, 2937 (1928).



In such cases exact determination was made by then condensing the water in the bulb, E, which was sealed off and weighed. The apparatus had a cluster of six or eight bulbs here, instead of one as illustrated, which could be replaced as desired by raising the proper traps.

The apparatus as indicated was connected to a system of pumps and to a reservoir of air-free water. The McLeod gage, the bulb of hydrate, the manometer, and the trap, C, were all immersed in the water of a thermostat, the mercury leads extending through the bottom to atmospheric height. At  $35^{\circ}$  the temperature variation of the thermostat was  $0.005^{\circ}$ . The temperature was read off a Beckmann thermometer which was calibrated against a thermometer certified by the Bureau of Standards. The latter had scale divisions of  $0.01^{\circ}$ .

The measurements of the pressure were taken by means of a cathetometer, using a millimeter scale set in brass.

The mercury used was cleaned by running it in a thin stream successively through 1500 centimeter columns of dilute nitric acid and distilled water. It was then distilled in vacuo.

The cupric sulfate used was Baker's C. P. recrystallized a number of times from distilled water.

Approximately 12 grams of the pentahydrate of cupric sulfate was used for each sample in the experiments described.

### Deaeration

Preliminary experiments on eliminating air showed that it was possible to get the salt in a condition where the amount of permanent gas in the vapor phase was very small, while there was still a considerable amount retained by the solid. The final procedure adopted for deaeration was as follows. The weighed sample of the pentahydrate of cupric sulfate was placed in the bulb, A, and the system was pumped out as far as possible. The treatment of the salt was essentially a succession of hydrations and dehydrations. The pentahydrate was heated at  $250^{\circ}$ , the water being pumped out as it appeared, until the pressure above the salt remained constant at  $10^{-4}$  millimeter, when dehydration was considered complete. According to Krafft<sup>5</sup> hydrated cupric sulfate loses its water in vacuo completely at  $250^{\circ}\text{C}$ . The salt was then rehydrated by opening to air-free water while the bulb was surrounded by ice. The dehydration was then repeated.

At first the salt was hydrated completely before each withdrawal of water. However, a series of experiments showed that the greatest part of the permanent gases appeared only during the loss of the last mol or so of water, that is, when heating from  $150^{\circ}$  to  $250^{\circ}$ . For this reason complete hydration was discontinued. During the latter part of the process the anhydrous salt at room temperature was thrown in contact with the reservoir of air-free water and allowed to hydrate over a period of from two to four hours. The manifold was then trapped, after being tested for air-free conditions; the salt was left open to the manifold. The temperature around the bulb of cupric sulfate was raised slowly to  $250^{\circ}$ , the water driven off condensing in the manifold. When

<sup>5</sup> Krafft: Ber., 40, 4770 (1907).

the outflow of water had ceased, the vapor above the liquid water in the manifold was examined for air. The water in the manifold was then pumped out and the whole system, including the bulb of salt at  $250^{\circ}$ , was pumped down to  $10^{-4}$  millimeter. The pumping was maintained at this pressure for two or three hours.

Such a succession of operations was run on the cupric sulfate for periods of from two to three weeks. When the amount of air appearing in the manifold at the end of a dehydration was of the order of  $5 \times 10^{-4}$  to  $10^{-3}$  millimeter conditions were considered as satisfactory for undertaking measurements.

### The Measurements

*Measurements of dissociation pressure above the systems  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ — $\text{CuSO}_4 \cdot 3\text{H}_2\text{O}$  and  $\text{CuSO}_4 \cdot 3\text{H}_2\text{O}$ — $\text{CuSO}_4 \cdot \text{H}_2\text{O}$ .* The pentahydrate of cupric sulfate was taken as the starting point for the measurements. This was secured by condensing successive portions of air-free water on the salt, after determining the amount of this water by condensing it first in the capillary, D, Fig. 1. Sufficient water was run in on the salt to insure an excess above that equivalent to five mols per mol of cupric sulfate.

The salt was left standing for four days in contact with this excess water to insure complete hydration and also with the view of seeing how permanent gas accumulated during this time. In four days approximately  $1.5 \times 10^{-4}$  millimeter of air appeared in the manifold. During this period parts of the system had been as hot as  $70^{\circ}$  due to work with the thermostat. It was thought that a good portion of this air might have come out of the glass walls.

The pentahydrate was finally secured by having the bath around the bulb of salt and excess water regulating at  $35^{\circ}$ . The dissociation pressure of the hydrate at this temperature is 16.4 millimeters. The vapor pressure of a saturated solution of cupric sulfate at  $35^{\circ}$  is about 33 millimeters. The room temperature was around  $26^{\circ}$ . By leaving the trap, C, open, it is clear that distillation of any water in the bulb would occur into the manifold at room temperature. The vapor pressure of the condensed water would prevent the dissociation of any hydrate.

When obtained, the pentahydrate was warmed until the desired amount of water was lost, this being condensed in the capillary, D, and then in a small bulb which was sealed off and weighed. The method of simply heating the hydrate to drive out water had to be subjected to further control. Several methods were attempted. The one finally adopted consisted in heating the hydrate to such a temperature that its dissociation pressure was in excess of the vapor pressure of water at the temperature of the manifold. The water would then condense in the manifold as it was lost by the hydrate. The amount to be withdrawn was condensed in a bulb, sealed off, and weighed. The temperature of the hydrate was then lowered to such an extent that the vapor pressure of the water remaining in the manifold was in excess of the dissociation pressure of the particular hydrate pair desired. That is, in working with the system  $\text{CuSO}_4 \cdot 3\text{H}_2\text{O}$ — $\text{CuSO}_4 \cdot \text{H}_2\text{O}$ , conditions were so adjusted that there could be no formation of  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ .



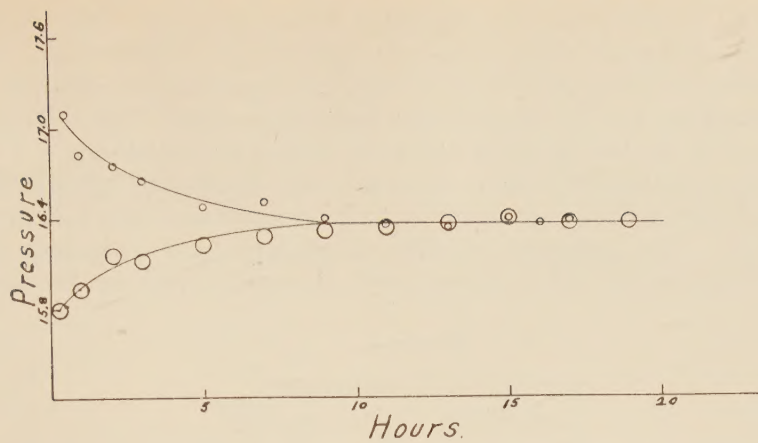


FIG. 2

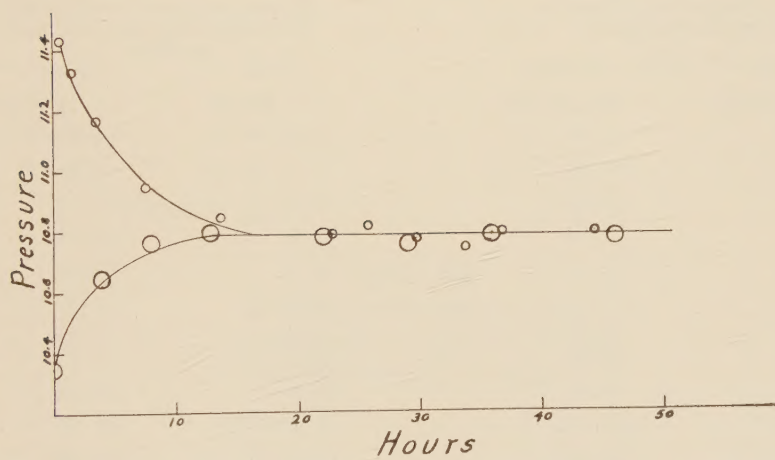


FIG. 3

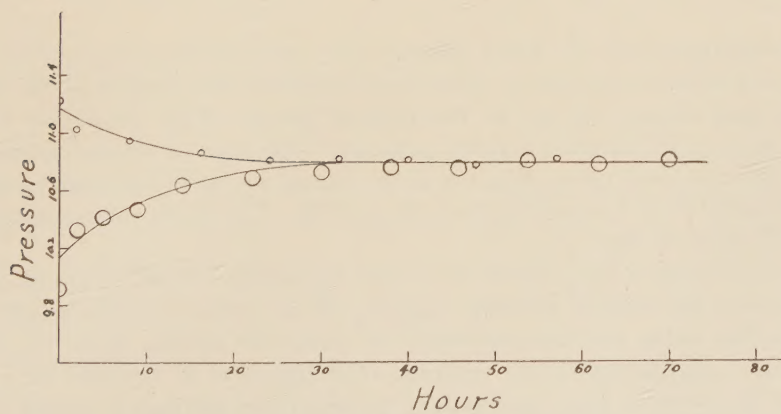


FIG. 4

The same considerations were applied in driving water out. This process of partial hydration and dehydration was repeated two or three times. The measurements available in the literature were used in making the adjustments. The pentahydrate was not reformed after each measurement. The additional water necessary to alter the water content as desired was withdrawn.

Measurements from the high pressure side were obtained by introducing a pressure of water vapor into the manifold in the desired excess over that in the hydrate bulb. The manometer was then drawn down until the hydrate was open to this excess, the trap, C, being raised. By such an operation the excess

TABLE I

Summary of Measurement 3  
Composition  $\text{CuSO}_4 \cdot 3.67 \text{H}_2\text{O}$   
Temperature  $35^\circ \pm 0.005$

| a.<br>Excess pressure of 9 millimeters<br>expanded onto hydrate |           | b.<br>Excess pressure of 7 millimeters<br>in hydrate bulb expanded into<br>manifold |           |
|-----------------------------------------------------------------|-----------|-------------------------------------------------------------------------------------|-----------|
| Time                                                            | Pressure  | Time                                                                                | Pressure  |
| 0.0 Hours                                                       | 18.35 mm. | 0.0 Hours                                                                           | 15.13 mm. |
| 0.5 "                                                           | 17.10 "   | 0.3 "                                                                               | 15.80 "   |
| 1.0 "                                                           | 16.83 "   | 1.0 "                                                                               | 15.93 "   |
| 2.0 "                                                           | 16.76 "   | 2.0 "                                                                               | 16.17 "   |
| 3.0 "                                                           | 16.65 "   | 3.0 "                                                                               | 16.13 "   |
| 5.0 "                                                           | 16.48 "   | 5.0 "                                                                               | 16.24 "   |
| 7.0 "                                                           | 16.52 "   | 7.0 "                                                                               | 16.30 "   |
| 9.0 "                                                           | 16.41 "   | 9.0 "                                                                               | 16.33 "   |
| 11.0 "                                                          | 16.38 "   | 11.0 "                                                                              | 16.35 "   |
| 13.0 "                                                          | 16.36 "   | 13.0 "                                                                              | 16.38 "   |
| 15.0 "                                                          | 16.42 "   | 15.0 "                                                                              | 16.42 "   |
| 16.0 "                                                          | 16.38 "   | 17.0 "                                                                              | 16.39 "   |
| 17.0 "                                                          | 16.40 "   | 19.0 "                                                                              | 16.39 "   |
| Mean                                                            | 16.39 "   | Mean                                                                                | 16.38 "   |

pressure expanded into the lower pressure side, initially assuming four-fifths of what it was before expansion. After such expansion the mercury in the manometer was allowed to rise to the desired height. This procedure was simply reversed in following a rising pressure. The type of results obtained and the time required for equilibrium to be reached are best illustrated by the detailed sets of data shown in Tables I, II, and III. Curves for these measurements are shown in Figs. 2-4.

The time marked zero time in the tables represents a lapse of about ten minutes from the time of actually trapping off the hydrate under its initial pressure, this being the time necessary to pump the system down to  $10^{-4}$  millimeter. The pressures are recorded in millimeters of mercury at  $35^\circ$ . Each is the mean of from two to four cathetometer settings in which the maximum deviation from the mean never exceeded 0.02 millimeter.



TABLE II

Summary of Measurement 5  
 Composition  $\text{CuSO}_4 \cdot 2.69 \text{ H}_2\text{O}$   
 Temperature  $35^\circ \pm 0.005$

| a.<br>Excess pressure of 5 millimeters<br>expanded onto hydrate |           | b.<br>Salt warmed up from $26^\circ$ . First<br>reading taken at $35^\circ$ |           |
|-----------------------------------------------------------------|-----------|-----------------------------------------------------------------------------|-----------|
| Time                                                            | Pressure  | Time                                                                        | Pressure  |
| 0.0 Hours                                                       | 11.86 mm. | 0.0 Hours                                                                   | 10.35 mm. |
| 0.3 "                                                           | 11.43 "   | 4.0 "                                                                       | 10.65 "   |
| 1.3 "                                                           | 11.33 "   | 8.0 "                                                                       | 10.76 "   |
| 3.3 "                                                           | 11.17 "   | 13.0 "                                                                      | 10.80 "   |
| 7.3 "                                                           | 10.95 "   | 22.0 "                                                                      | 10.78 "   |
| 14.3 "                                                          | 10.85 "   | 29.0 "                                                                      | 10.76 "   |
| 23.3 "                                                          | 10.79 "   | 36.0 "                                                                      | 10.79 "   |
| 26.3 "                                                          | 10.82 "   | 46.0 "                                                                      | 10.79 "   |
| 30.3 "                                                          | 10.78 "   | Mean 10.78 "                                                                |           |
| 34.3 "                                                          | 10.75     |                                                                             |           |
| 37.6 "                                                          | 10.80     |                                                                             |           |
| 47.0 "                                                          | 10.79     |                                                                             |           |
| Mean 10.79 "                                                    |           |                                                                             |           |

TABLE III<sup>6</sup>

Summary of Measurement 8  
 Composition  $\text{CuSO}_4 \cdot 1.09 \text{ H}_2\text{O}$   
 Temperature  $35^\circ \pm 0.005$

| a.           |           | b.           |          |
|--------------|-----------|--------------|----------|
| Time         | Pressure  | Time         | Pressure |
| 0.0 Hours    | 11.22 mm. | 0.0 Hours    | 9.92 mm. |
| 2.0 "        | 11.03 "   | 2.0 "        | 10.33 "  |
| 8.0 "        | 10.95 "   | 5.0 "        | 10.41 "  |
| 16.0 "       | 10.86 "   | 9.0 "        | 10.47 "  |
| 24.0 "       | 10.81 "   | 14.0 "       | 10.64 "  |
| 32.0 "       | 10.82 "   | 22.0 "       | 10.69 "  |
| 40.0 "       | 10.81 "   | 30.0 "       | 10.73 "  |
| 48.0 "       | 10.78 "   | 38.0 "       | 10.77 "  |
| 57.0 "       | 10.82 "   | 46.0 "       | 10.76 "  |
| Mean 10.81 " |           | 54.0 "       | 10.81 "  |
|              |           | 62.0 "       | 10.78 "  |
|              |           | 70.0 "       | 10.81 "  |
|              |           | Mean 10.78 " |          |

<sup>6</sup> This measurement was obtained from a different sample of cupric sulfate. This is discussed in the summary.

*Measurements on the system  $\text{CuSO}_4 \cdot \text{H}_2\text{O}$ — $\text{CuSO}_4$ .* The variation of water content was brought about differently to obtain these measurements. On the first trials the cupric sulfate was completely dehydrated. An amount of water corresponding to 0.1 mol was condensed in the capillary, D. This was surrounded with ice. The dehydrated salt at a temperature of  $35^\circ$  was put in contact with this water. The pressure of the water vapor was thus such as to eliminate the possibility of any formation of trihydrate. The water was determined by condensing the same amount into a bulb and weighing.

A measurement was obtained on this mixture of monohydrate and anhydrous salt and then additional water was run in as already described above, the process being repeated until three measurements were obtained. These

| TABLE IV            |                           |               |
|---------------------|---------------------------|---------------|
| Duration of Heating | Temperature               | Mean Pressure |
| 5 Days              | $100^\circ$ — $110^\circ$ | 0.31 mm.      |
| 5 "                 | "                         | 0.21 "        |
| 6 "                 | "                         | 0.17 "        |
| 3 "                 | "                         | 0.17 "        |

showed a rising pressure as the water content increased. It was thought that this variation might be due to adsorbed water building up a pressure in excess of the dissociation pressure of the system. The reversion to the monohydrate would then be exceedingly slow at  $35^\circ$ , as indicated by the fact that the measurements obtained held constant over considerable periods of time.

The measurements were accordingly repeated. In this repetition the hydration was carried out as outlined above except that the salt was kept at a much higher temperature. Such temperature was maintained for several days after the water had been taken up. The salt was then cooled down slowly and a measurement obtained at  $35^\circ$ . Then the heating was resumed for a period, after which another measurement at  $35^\circ$  was made. This was continued until the measurements obtained after such periods of heating checked. For the two measurements on the monohydrate at the lower water content, the temperature of heating was  $100^\circ$  to  $110^\circ$ . With the higher water content it was necessary to heat at  $140^\circ$  to  $150^\circ$  in order to get equilibrium in any reasonable length of time.

The general course of the measurements is illustrated by Table IV which shows a summary of the measurement on  $\text{CuSO}_4 \cdot 0.126 \text{H}_2\text{O}$ .

In obtaining the mean pressures recorded above and also in the final summary, measurements were run from both sides of equilibrium. To measure a rise from the lower side, the pressure above the hydrate, 0.18 mm., was allowed to expand into the manifold, the manometer was raised and the system pumped out. By the time pressure readings could be taken they were, within the limits of error, 0.18 mm.

Measurements were also taken with a dropping pressure, but here the change was much more gradual and was such as to indicate that a rather large initial pressure resulted in some adsorption. For this reason the excess pressure allowed to expand onto the salt was usually 1 to 2 millimeters. This dropped to the equilibrium value in the course of 6 to 12 hours.



### Results

A summary of the measurements obtained is shown in Table V. The measurements evidently establish the existence of the penta-, tri-, and mono-hydrates of cupric sulfate.

The measurements are plotted in Fig. 5.

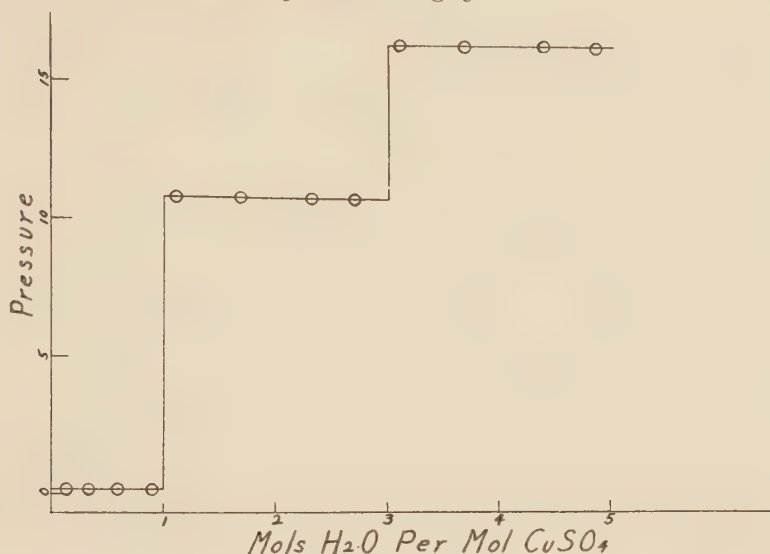


FIG. 5

TABLE V

| Number | Composition                               | Mean Pressure |
|--------|-------------------------------------------|---------------|
| 1      | CuSO <sub>4</sub> . 4.84 H <sub>2</sub> O | 16.39 mm.     |
| 2      | CuSO <sub>4</sub> . 4.41 H <sub>2</sub> O | 16.40 "       |
| 3      | CuSO <sub>4</sub> . 3.67 H <sub>2</sub> O | 16.39 "       |
| 4      | CuSO <sub>4</sub> . 3.10 H <sub>2</sub> O | 16.42 "       |
| 5      | CuSO <sub>4</sub> . 2.69 H <sub>2</sub> O | 10.79 "       |
| 6      | CuSO <sub>4</sub> . 2.32 H <sub>2</sub> O | 10.80 "       |
| 7      | CuSO <sub>4</sub> . 1.68 H <sub>2</sub> O | 10.80 "       |
| 8      | CuSO <sub>4</sub> . 1.09 H <sub>2</sub> O | 10.80 "       |
| 9      | CuSO <sub>4</sub> . 0.91 H <sub>2</sub> O | 0.17 "        |
| 10     | CuSO <sub>4</sub> . 0.59 H <sub>2</sub> O | 0.18 "        |
| 11     | CuSO <sub>4</sub> . 0.34 H <sub>2</sub> O | 0.18 "        |
| 12     | CuSO <sub>4</sub> . 0.13 H <sub>2</sub> O | 0.17 "        |

In obtaining these results, two samples of cupric sulfate were used. The first seven measurements were obtained with the first sample, the last five with the second. During the course of the experiments on the first sample, breaks in the apparatus admitted air on two occasions. This required that the process of deaeration be repeated twice after the first time. Measurements 1 and 6 were obtained after the first deaeration; 2, 3, and 4 after the second; 5 and 7 after the third. After Measurement 7 was obtained the salt

was accidentally heated to a temperature of about  $325^{\circ}$ . Some decomposition occurred and it was found to be unsuitable for further work. It was accordingly discarded.

In working with the second sample it was found that heating to about  $110^{\circ}$  and at the last to  $150^{\circ}$  caused the loss of four mols of water, and that then it was possible to pump the system down to  $10^{-4}$  millimeter while keeping the salt at a temperature of  $100^{\circ}$ . Over a period of four hours this pressure increased to about  $2 \times 10^{-3}$  with no pumping and the amount of permanent gas present was very small. This was considered as indicative that air-free conditions could be maintained at a lower temperature and a higher water content.

The salt was hydrated completely and enough water withdrawn to insure its being a mixture of the two higher hydrates. Two measurements were then obtained at  $35^{\circ}$  one from each side of equilibrium. The values were 16.38 and 16.40 millimeters. The salt was then subjected to treatment previously outlined to reduce its composition to  $\text{CuSO}_4 \cdot 1.09 \text{H}_2\text{O}$ .

Measurement 8 was thus obtained without removal of such deep-seated air as had been taken out in the previous cases. Such a course of complete deaeration was however gone through, before the measurements on the monohydrate were taken. At the end of these measurements the salt was again hydrated completely and then brought to a composition of about  $\text{CuSO}_4 \cdot 4.5 \text{H}_2\text{O}$ . A measurement of the dissociation pressure at  $35^{\circ}$  was in satisfactory accord with those obtained earlier.

At the end of each measurement the water vapor above the hydrated salt was examined for permanent gas. In no case did the partial pressure of this exceed  $2 \times 10^{-3}$  millimeter, and in most cases it was considerably less than this. The data tabulated have shown that the absence of permanent gas has a very marked effect on the time required for equilibrium to be reached. In the case of the highest hydrate pair equilibrium was reached with considerable rapidity—about an hour as compared with much longer periods mentioned elsewhere. In the case of the lower pairs the process is slower, but still fairly rapid.

### Summary

1. An apparatus is described for the determination of the dissociation pressures of hydrated salts by the statical method.
2. A method for eliminating adsorbed air from such hydrates is described.
3. The  $35^{\circ}$  isotherm for the dissociation pressures of cupric sulfate containing a varying amount of water of hydration has been determined under air-free conditions.
4. The measurements obtained indicate the existence of  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ,  $\text{CuSO}_4 \cdot 3\text{H}_2\text{O}$ , and  $\text{CuSO}_4 \cdot \text{H}_2\text{O}$ .

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### BIOGRAPHY

The author was born in Hogansville, Georgia, on April 13, 1903. He received his secondary education at various schools in Georgia, graduating from the Elberton High School in the spring of 1920. He attended the Georgia School of Technology for a year, then transferred to Emory University. He received the degree of Bachelor of Science from the latter in 1924 and the degree of Master of Science in 1925. During the years 1925-1926-1927 he was Instructor in Chemistry at the South Georgia Agricultural and Mechanical College. He entered the Johns Hopkins University in the fall of 1927 and has since been engaged in graduate study there.













